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RIGID CLOSED-CELL POLYIMIDE FOAMS FOR AIRCRAFT APPLICATIONS AND FOAM-IN-PLACE TECHNOLOGY

Final Report

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by

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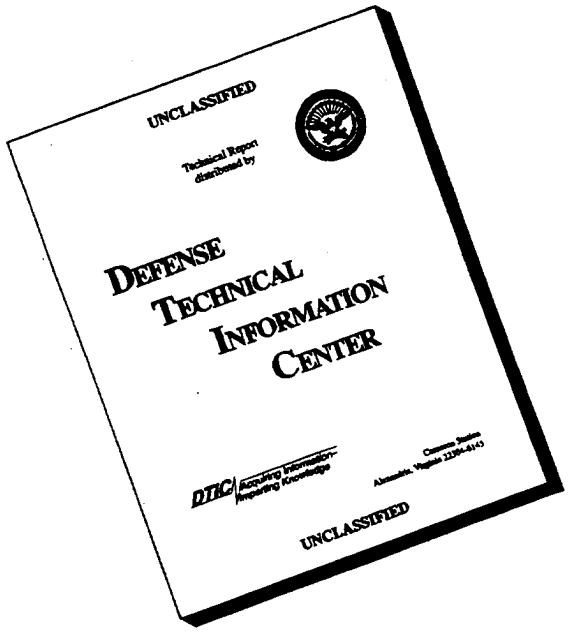

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Part I

ABSTRACT

The following paragraphs contain a summary of the significant accomplishments generated during the course of the contract effort.

- Part I, which covers the work dealing with testing of closed-cell foams, has resulted in the characterization of compositions which produce rigid foams for use in galley structure applications.

- The same effort has shown that the density, compressive strength and shear strength of the foams are directly related to the concentrations of the microballoons. The same properties are also directly related to the resin loading.

- Prototype samples of rigid closed-cell foams meeting the requirements of the program have been submitted to NASA, LB Johnson Space Center.

- Part II, which was undertaken to investigate apparatus to produce polyimide foams using foam-in-place techniques, has resulted in the selection of a spray gun apparatus capable to deliver a mixture of microballoons and resin binder on substrates which cures to yield a closed-cell foam.

- The adhesion of the foam on aluminum, titanium and steel substrates has been found to be excellent.

- The material meets the mechanical and thermal requirements of the program.

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PROGRAM SCOPE AND OBJECTIVE

This program is divided into two parts. Part I covers the work dealing with characterization of closed-cell foams for use in aircraft applications and Part II covers the development of a spray apparatus to foam-in-place.

PART I

This program has been undertaken to investigate, characterize and select closed-cell, rigid, foams for use in cargo bay floor panels and galley structures of commercial aircraft. The major objective of this program was the fabrication of these materials in shape and size for replacement of more flammable products presently used in aircraft interiors.

Part I of this program consists of six tasks which define the general objectives of the Work Plan.

The Work Plan constituting the various tasks of the program and their milestones is shown in Figure 1.

Task I covers the study of the optimum parameters including BTDA-oxoimine ratio which favor foams with the highest closed-cell content and best mechanical properties.

Task II is an evaluation of the aromatic diamine component. The selection of the diamine is done by taking into consideration its effects on thermal properties of the foam, associated health problems and cost.

Task III is the study of processes to produce closed-cell foams.

Task IV covers the processes to produce closed-cell foams by syntactic methods.

The various candidates were evaluated in Task V followed by selection of the optimum material which was scaled-up to provide samples for evaluation by NASA-LB Johnson Space Center (Task VI).

A final task dealing with Reporting and Coordination covers the effort necessary to report the program status and includes a midterm and one final presentation to acquaint NASA-LB Johnson Space Center technical personnel with the progress of the program.

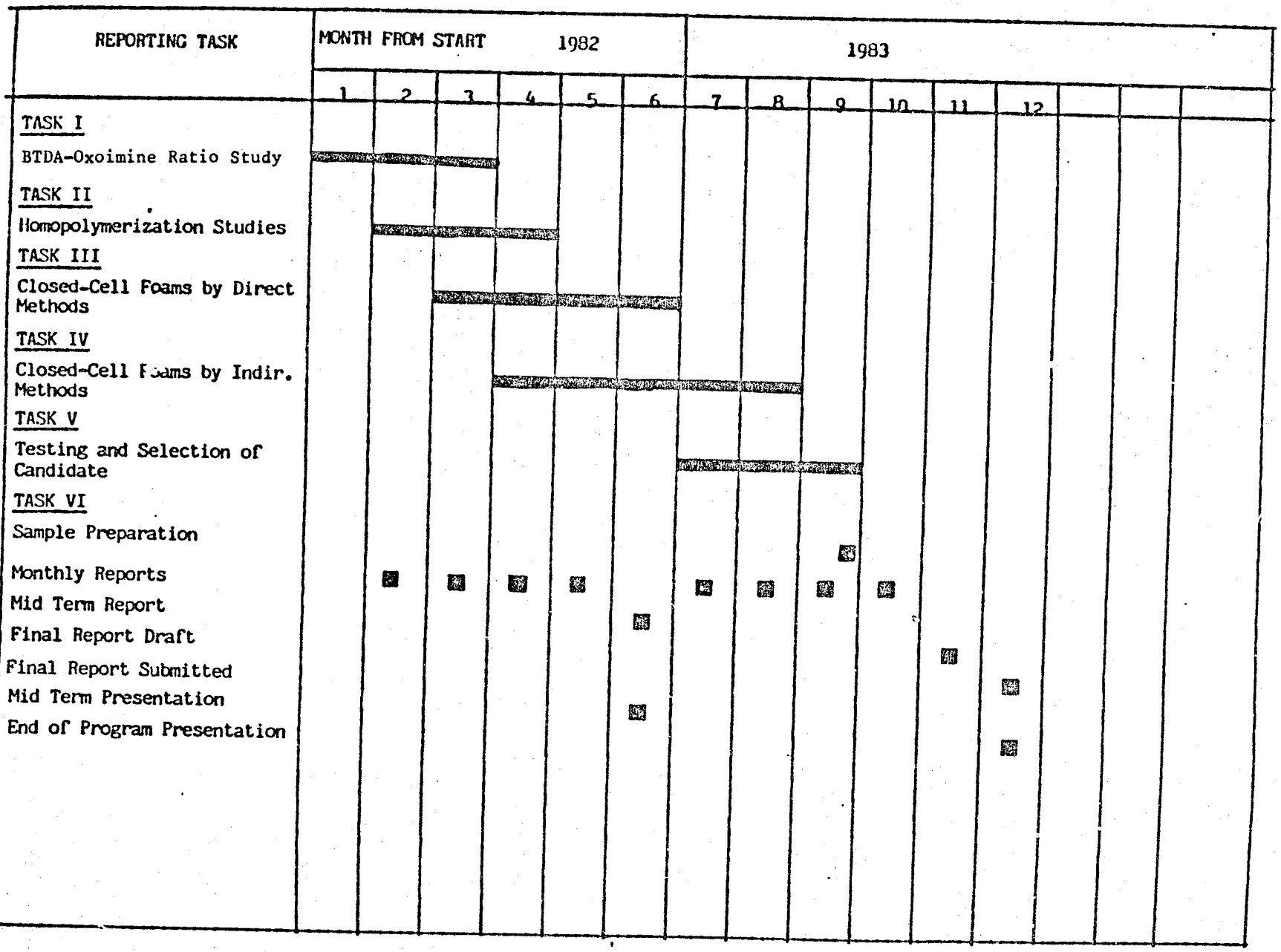

Figure 1 - PROGRAM SCHEDULE

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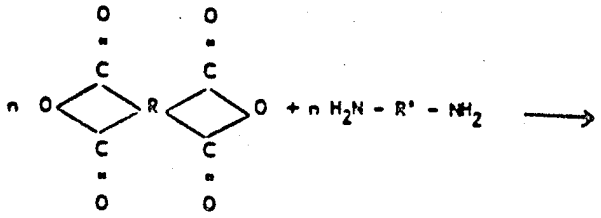
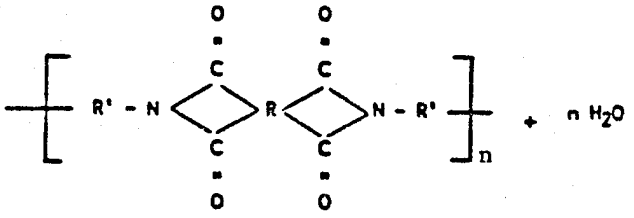
BACKGROUND AND PROGRAM APPROACH

2.1 Discussion of the Proposed Program - General Approach

Conventional polyimide resins have been prepared by a method which consists of reacting one or more aromatic diamines with a dianhydride in an inert solvent. This process produces a polyamic acid solution which, after evaporation of the solvent and application of additional heat undergoes a condensation reaction with complete imidization and formation of a high molecular weight polyimide structure as shown below:

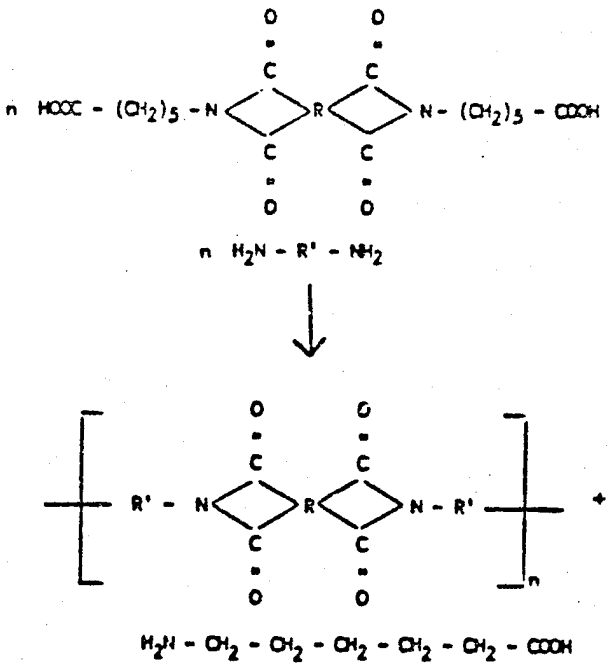




In a second method of preparing conventional polyimide resins the dianhydride is reacted with an alcohol to form a diester to which the diamine or diamines are added. Evaporation of the alcohol followed by application of heat produces a high molecular weight polyimide resin similar to that shown before. The two methods yield the same polymer, but only the second method produces foams. The polyimide foams obtained from this type of composition posses an open-cell structure which is flexible and resilient and finds use in a variety of spacecraft and aircraft applications (Ref. 1, 2, 3, 4).

Chem-tronics' novel polyimide technology offers a new method for processing and foaming polyimide resins by the use of a unique reaction scheme which yields foams with a closed-cell structure.

These novel polyimides are synthesized by first reacting a dianhydride such as 3,3', 4,4' benzophenonetetracarboxylic acid dianhydride, (BTDA), with an oxoimine. This reaction produces a carboxy terminated bisimide monomer which is further reacted with aromatic diamines in an inert or reactive solvent and polymerized to an imide by an exchange reaction as shown in the following scheme.



Various types of solvents can be used in the Chem-tronics' process to yield different finished products.

Specially formulated liquid resins made by the Chem-tronics process, when further treated by evaporation of the solvent, give free flowing powders stable at room temperature. These powders when heated in an oven at specified temperature are converted into foams possessing a closed-cell structure (Ref. 5).

The cell structure of the foam is controlled by the reactivity of the starting powder and by the processing conditions. When the condensation reaction is carried out at a temperature in the range of 148.9°C (300°F) the powder resins foam and cure without excessive formation of volatiles. The cell walls of foams produced at such a low temperature have a tendency to stretch rather than break, and closed-cell foams are obtained.

The technology to produce closed-cell foams is available in the Chem-tronics Research Laboratories on a pilot plant scale.

The following program outlines approaches to the fabrication of closed-cell, modified polyimide foams from specially synthesized resins followed by selection of a candidate meeting the requirements for use in cargo bay and galley panels.

3

EXPERIMENTAL PROCEDURES

This section outlines the experimental procedures used in the preparation of the materials evaluated in this program and includes synthesis of the liquid and powder resins (Section 3.1), closed-cell foams by direct methods (Section 3.2) and closed-cell foams by indirect methods (Section 3.3).

3.1 Synthesis of the Liquid Polyimide Resins and Powder Polyimide Resins

Benzophenonetetracarboxylicacid dianhydride (BTDA) and the oxoimine reaction product were added to 200 ml of alcohol and refluxed until clear. The mixture was cooled to 40-50°C (104-122°F) and the diamine added. The mixture was then stirred until clear and allowed to cool.

The mixture was then transferred to 61 x 91 cm steel dishes (24 x 36 inch) and placed in a circulating air oven preheated at 76.6°C (170°F). The resin was maintained at these conditions for 10-12 hours.

The solid mass was then passed through a grinder and sifted. The powder left on U.S. No. 20 screen (opening of 0.84 mm, 0.331 inch) was collected and stored for further processing.

3.2 Closed-Cell Polyimide Foam by Direct Methods

The powder resin obtained as described in Section 3.1 was placed in a closed mold and foamed in a microwave oven at a power of 6kW for 10 minutes. The powder expanded and formed a well consolidated structure having a density of 32-80 kg/m^3 (2-5 lb/ft^3).

3.3 Closed-Cell Polyimide Foams by Indirect Methods

Indirect methods of fabrication of closed-cell foams, also known as syntactic methods, are carried-out by adhesively bonding microballoons with a resin binder.

In this process, the balloons are mixed with the liquid resin and the mixture placed in a mold to convert the binder to a fully cured polymer.

Initial tests included visual observation, followed by determination of density, compressive strength and shear strength.

Compression and shear tests were carried out using the procedure described in Mil-Std-401.

The apparatus used was an Instron, Model 1000 with a full scale load range of454 kg (1000 lb) and a crosshead speed range of 0.01 to 50 cm/minute.

4

EXPERIMENTAL RESULTS

This section covers the work carried out to characterize and select closed-cell rigid foams meeting the requirements for cargo bay floor and galley core materials and starts with studies of the reaction (Section 4.1) followed by homopolymerization studies (Section 4.2) closed-cell foams by direct methods (Section 4.3) and closed-cell foams by indirect methods (Section 4.4).

4.1 Task I - BTDA-Oxoimine Reaction Product Study

The objective of this task was to evaluate the effect of the BTDA-oxoimine reaction product on the mechanical and physical properties of rigid closed-cell foams.

The balloons obtained from these resin compositions were evaluated for density, homogeneity of size and yield. Figure 4-1 shows the effect on density. The density of the balloons decreases almost linearly as the BTDA oxoimine reaction product increases, but it remains practically constant at values of 0.2 moles and higher.

The foaming process was carried out by placing the powder resin in an aluminum mold followed by heating at a sufficiently high temperature to cause expansion of the powder resin.

The results obtained from this study, are shown in Figure 4-1.

4.2 Task II - Homopolymerization Studies

The major objective of this task involves the study of compositions prepared with different aromatic diamines. These diamines were selected taking into consideration their effect on the thermal properties of the materials and cost.

Because of these limitations only the following aromatic diamines were evaluated:

p. phenylene diamine
m. phenylene diamine
4,4 diaminodiphenyl sulfone
4,4' diaminodiphenyl ether
2,6 diamino pyridine
2,4 toluene diamine
4,4' diaminodiphenyl methane

The resin compositions under study in this task were prepared using the process reported in Section 3.1.

The only diamines which were compatible with the process were, 4,4' diaminodiphenyl methane and 2,4-toluene diamine.

Because of availability, cost and ease of processing, 4,4'-diaminodiphenyl methane was selected as the optimum diamine for preparation of the resins and foams studied in this program.

In this task attempts were also made to optimize the quality of the balloons with the addition of surfactants to the liquid resins.

These additives are known to lower the surface tension of the growing bubbles and help prevent film rupture.

The surfactants tested were Fluorad 431 (3 M and Co.) and Zonyl FSC (DuPont) in concentrations of 0.51% to 1.0%, but they did not improve quality or yield of the product.

4.3 Task III - Closed-Cell Foams by Direct Methods

Microwave and thermal heating processes have been evaluated in this task.

To make a foam, the powder resin is laid in a mold and placed in a microwave cavity at a power output of 4-6 kW or in a thermal oven at a temperature of 204-232°C (400-450°F).

After a short period of exposure of 2-3 minutes, the powder expands into a homogeneous cellular material possessing essentially a closed-cell structure.

The mold configuration and material of construction depends upon the foaming process used.

The parameters that control the formation of closed-cell foams have been found to be: reactivity of the powder resins, powder loading, heating rate, and heating time.

The reactivity of the powder resin is a function of the the composition used. For the studies carried out in this task the resin compositions were prepared as discussed in Task I.

These powders produced foams with essentially a closed-cell structure which possessed a density characteristically in the range of 40 kg/m^3 (2.5 lb/ft^3) to 64 kg/m^3 (4.0 lb/ft^3) and a compressive strength in the range of 68.9-137.8 x 10^3 N/m^2 (10-20 psi). The values of compressive strength do not meet the minimum requirements for galley or cargo bay panels.

The mechanical properties of foams are generally improved by the addition of fillers including fibers, microballoons and minerals which are added to the powder or resin system.

The polyimide resins and the process used here, however, do not lend themselves to addition of reinforcements because they interfere with the foaming process and with the formation of a closed-cell structure.

Because the compressive strength properties of the foams produced by direct methods could not be readily improved within the scope of this program, the effort of this task was terminated.

4.4 Task IV - Closed-Cell Foams by Indirect Methods

These processes, commonly known as syntactic methods, yield closed-cell foams by adhesively bonding microballoons with a resin binder.

The process as carried out in this program yields closed-cell foams with a wide range of density characteristics.

The first task involved a study to determine the optimum cure temperature for the microballoons.

The density of the microballoons was found to decrease almost linearly from approximately 48 kg/m^3 (3 lb/ft^3) to 32 kg/m^3 (2 lb/ft^3) when the temperature was increased to 232.2°C (450°F).

It became evident from preliminary data that the microballoons alone offered little resistance to compressive forces. This property was important particularly for the cargo bay core material which requires a compressive strength of 4.1 x 10^6 N/m^2 (600 psi). Therefore, a study was initiated to evaluate other fillers in combination with the microballoons in an effort to improve this property.

Glass microballoons, type C15-250 produced by the 3 M Company were first evaluated by addition to the microballoons.

The effect of the glass microballoons on density and compressive strength of the closed-cell foams is shown graphically in Figures 4-2 and 4-3, respectively.

The shear strength of these same foams was found to be affected by the glass microballoon loading as shown in Figure 4-4.

The shear properties of syntactic foams, including the foams studied in this task, are dependent on the interfacial bond strength between each balloon and on the balloons surface to volume ratio.

The effects of weak interfacial strength are evident in Figure 4-4 which shows the relationship between shear strength of the syntactic foam and the glass microballoon concentration.

The shear strength of the syntactic foams is also dependent on the resin binder loading in the final product since binder loading contributes to higher interfacial bond strength.

This relationship is shown in Figure 4-5 where the shear strength of the syntactic foam increases almost linearly with an increase in the resin content. Similarly, the density and the compressive strength of the foams are dependent on the resin binder loading as shown in Figure 4-6 and Figure 4-7, respectively.

The density and compressive strength of the foams produced at the ratios shown increased linearly with an increase in the resin binder content in the final product.

At the conclusion of this work several compositions were selected for fabrication of prototype samples of galley core materials.

The finished foams possessed a density of 72-88 kg/m^3 (4.5-5.5 lb/ft^3), a compressive strength of 6.8-10.7 N/m^2 (100-120 psi) and a shear strength of 3.1-4.1 x 10^5 N/m^2 (45-60 psi). Prototype samples of two materials were submitted to Weber Aircraft for lamination and for determination of processability. The material was found to be compatible with presently used processes and met the critical requirement for compressive strength, shear strength and flammability. As reported previously these foams did not meet the specifications for cargo bay core materials.

A new method to produce cargo bay core materials was investigated by reinforcing low strength Nomex honeycomb having a cell size of 0.95 cm (0.375 inch) and a density of 48 kg/m^3 (3 lb/ft^3) with a resin followed by filling the cells with low density microballoons.

The use of short glass fibers blended in the resin was then investigated to optimize the compressive strength of the honeycomb at the lowest possible density. The effect of the glass fibers on the compressive strength of the honeycomb is shown in Figure 4-8.

As expected, the net effect of the addition of glass fibers to the resin was an increase of the density of the honeycomb which relates directly to the compressive strength as shown in Figure 4-9. Preliminary data also show that compressive strength values in the range of 11.0 x 10^6 N/m^2 (1600 psi) can be achieved at a density of approximately 192-240 kg/m^3 (12-15 lb/ft^3).

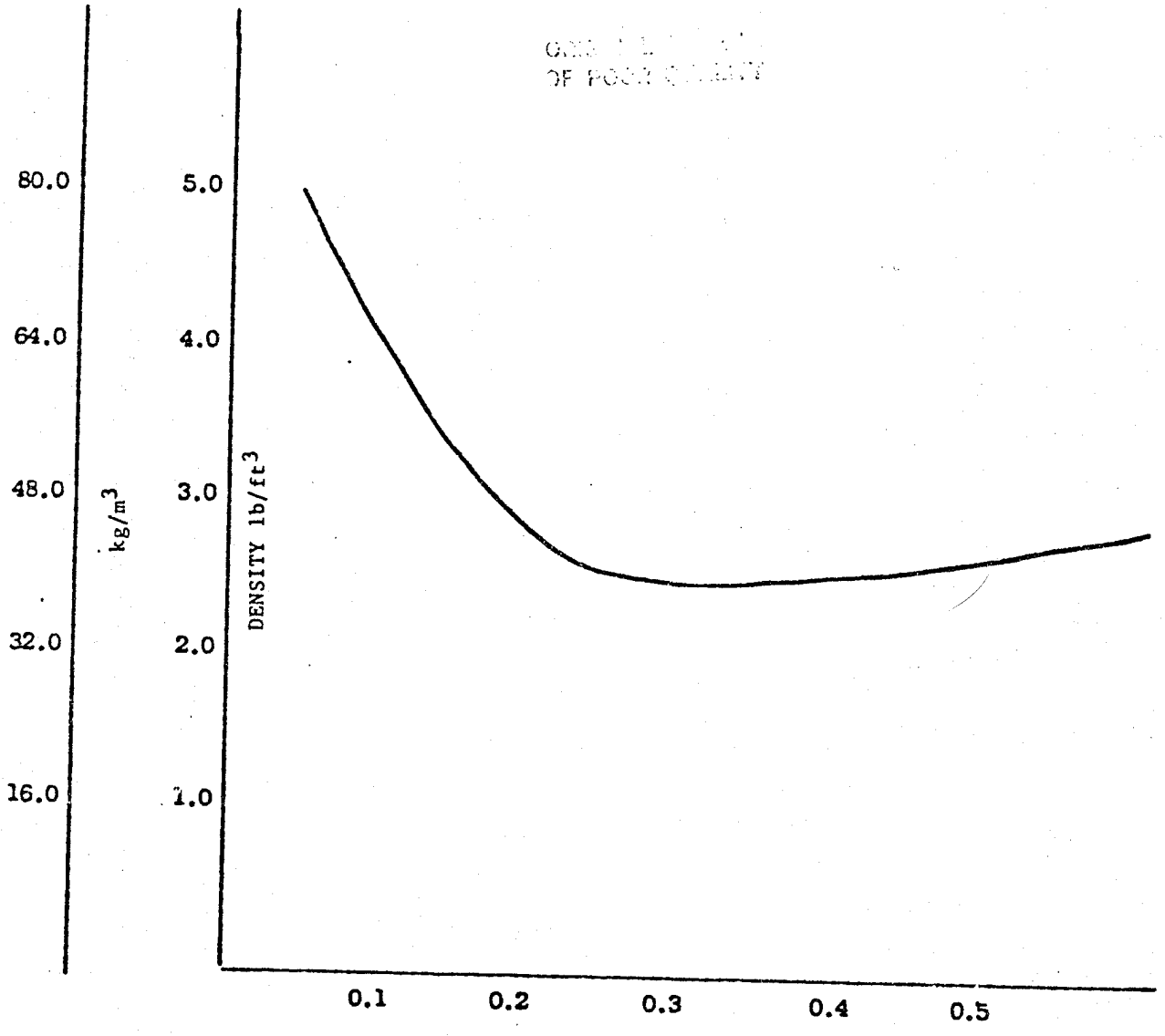
4.5 Task V - Testing and Selection

The objective of this task was to screen out potential candidates developed during the course of this program and to select at least one candidate core material meeting the requirements for galley and cargo bay core, respectively.

To fully identify the characteristics and to establish minimum functional requirements for each of the candidates identified testing was carried out from the start of the program for the most critical properties in accordance with the program goals.

The most critical properties for the galley core material were shear strength and compressive strength, respectively and density. The process employed in Task IV, previously discussed, provided a method to produce galley core materials meeting the requirements of the program.

Two of these candidates were submitted to Weber Aircraft for evaluation. The results obtained from Weber Aircraft are given in Table I.



BTDA-OXOIMINE REACTION PRODUCT, concentration

Figure 4-1

Effect of BTDA-oxoimine reaction product on density of microballoons.

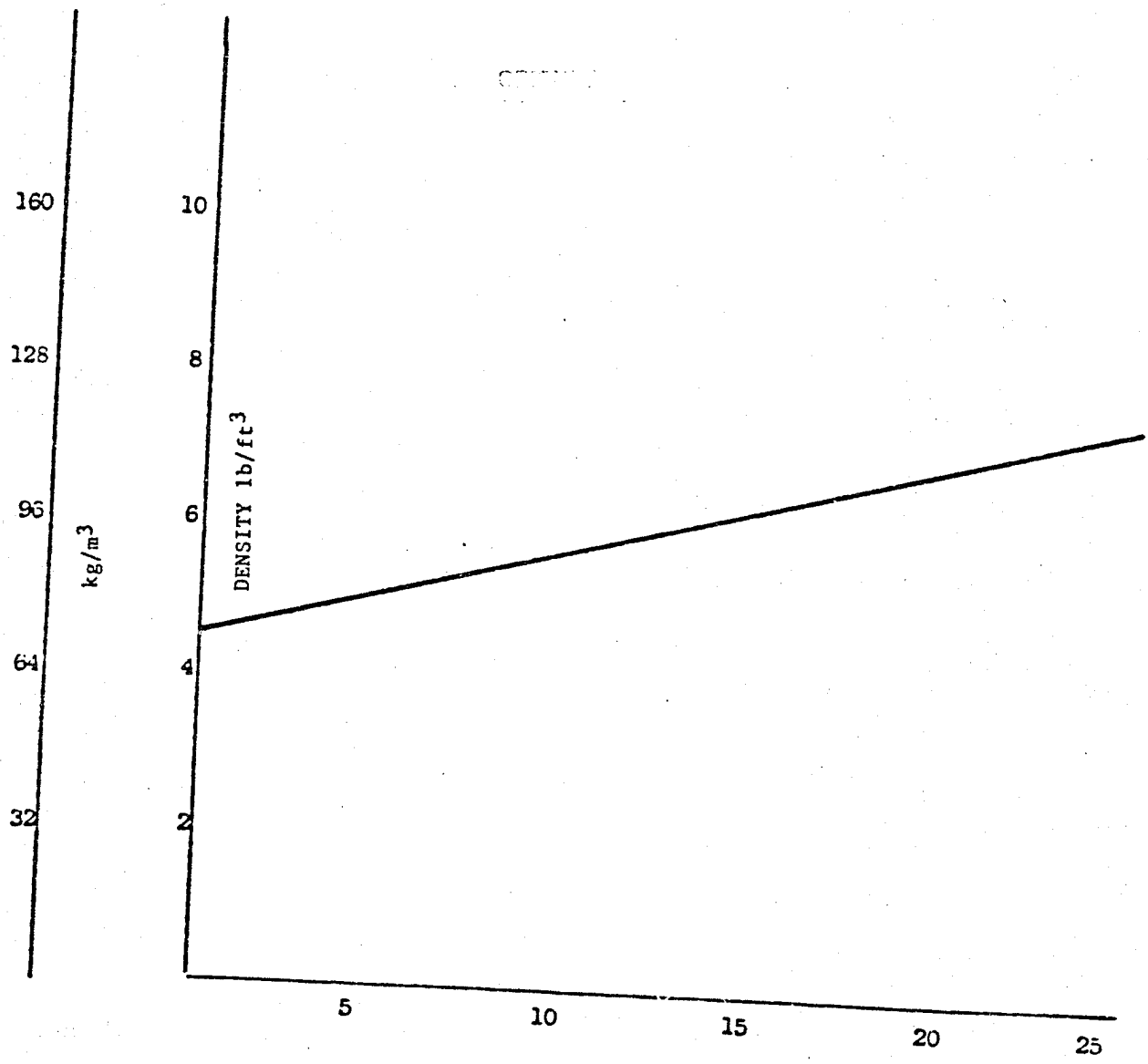


Figure 4-2

Effect of glass microballoon loading on the density of closed-cell foams.

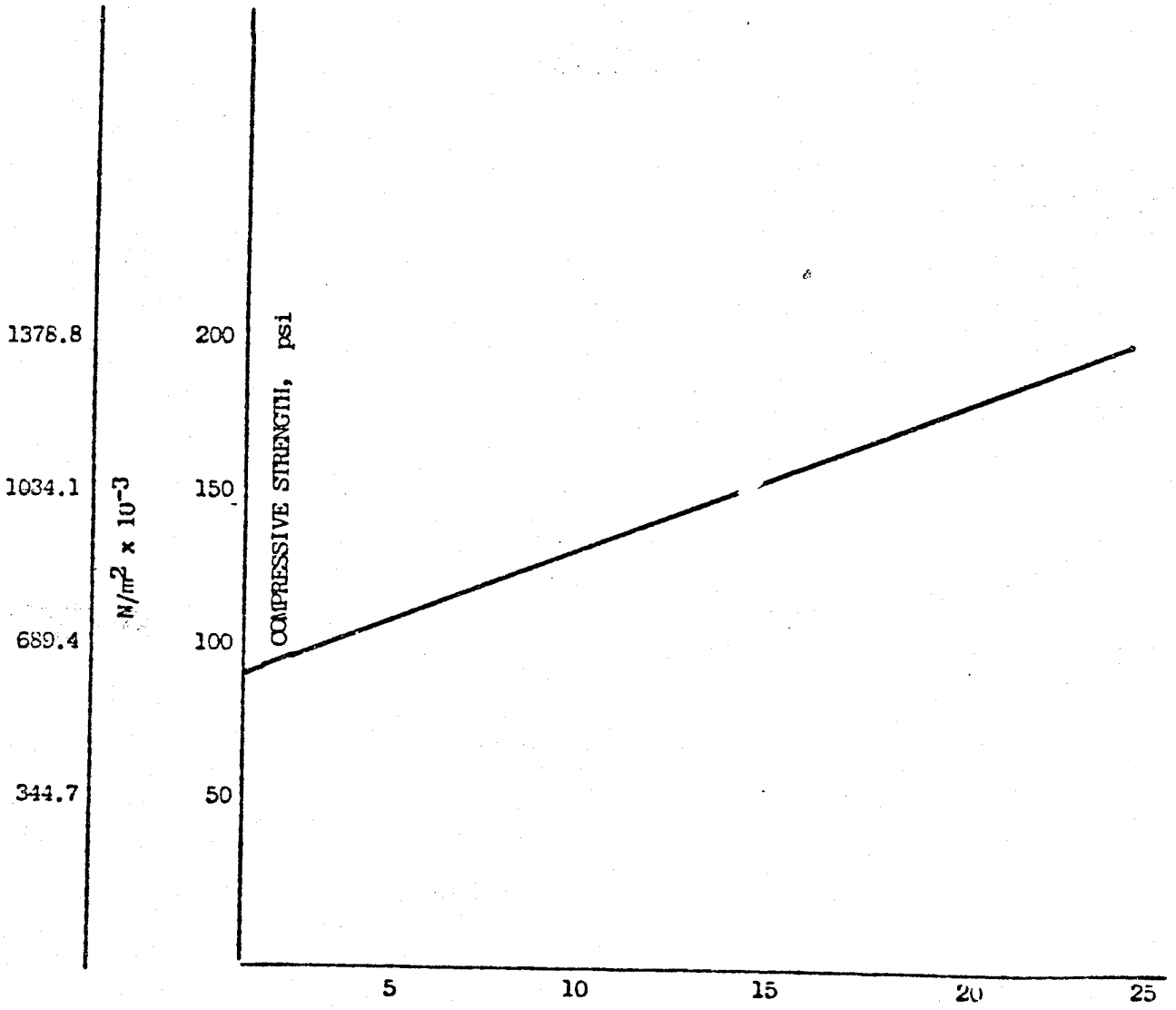


Figure 4-3

Effect of glass microballoon loading on the compressive strength of closed-cell foams.

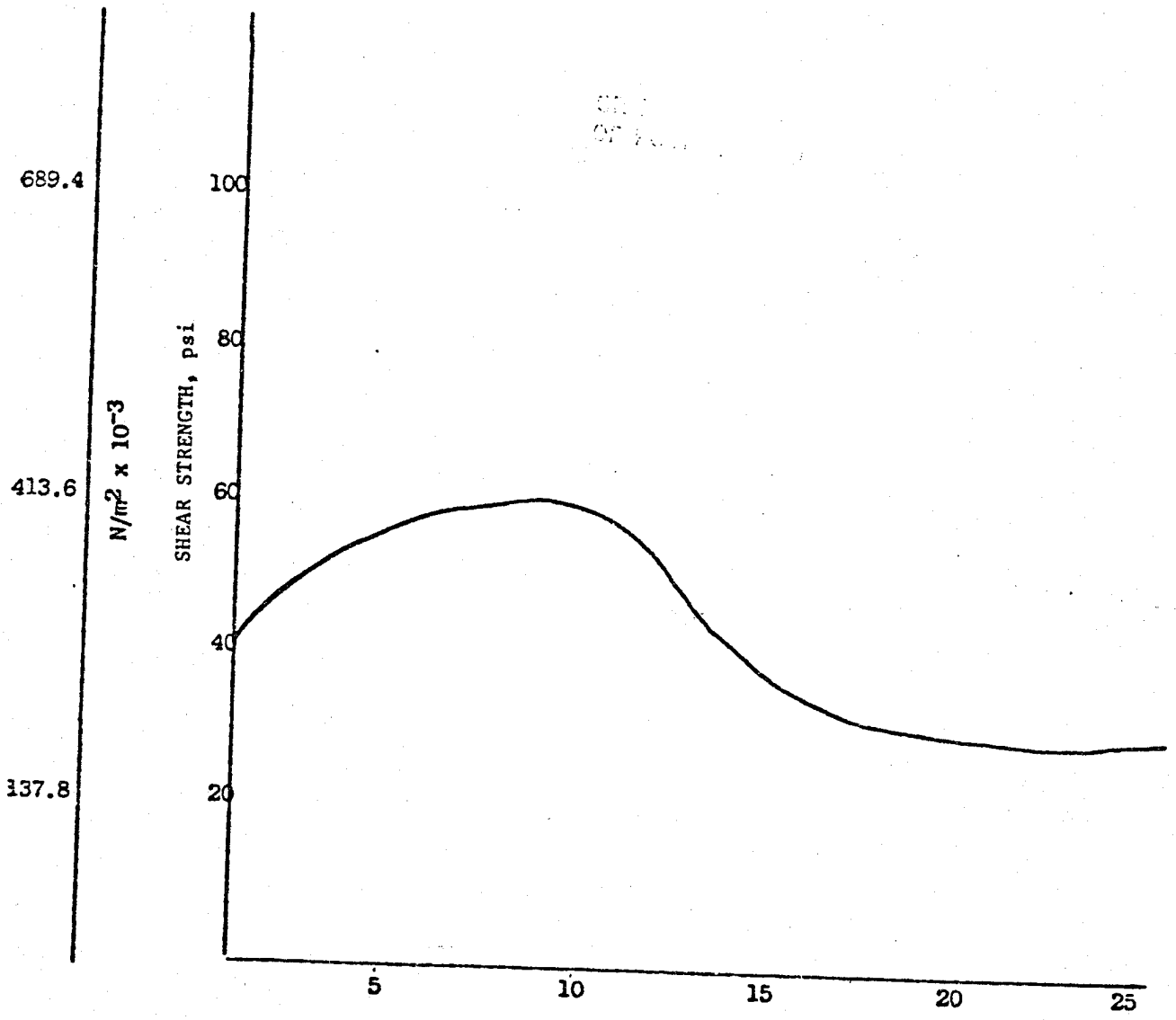


Figure 4-4

Effect of glass microballoon loading on the shear strength of closed-cell foams.

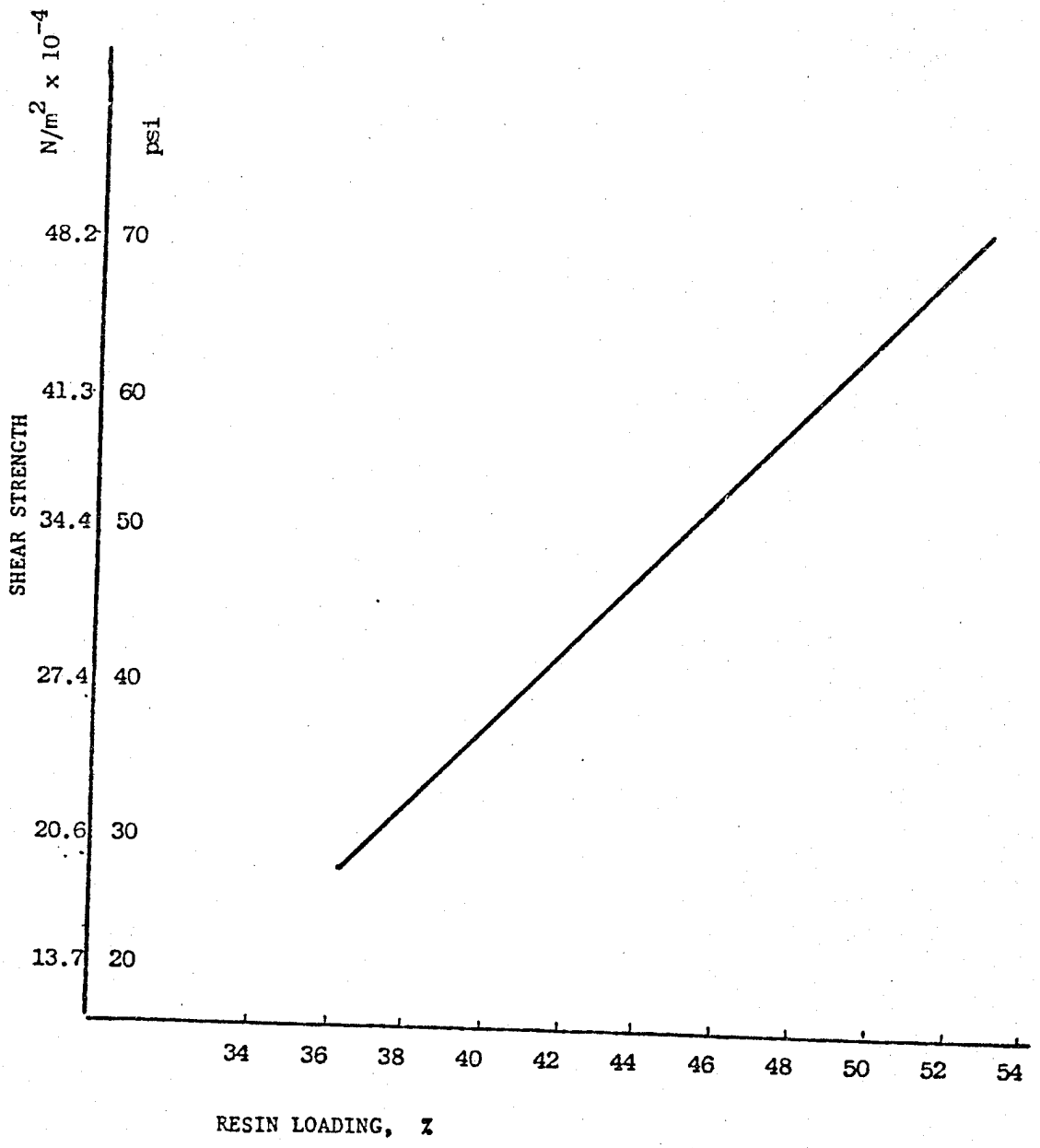


Figure 4-5

Effect of Binder Loading on the Shear Strength of Closed-Cell Foams.

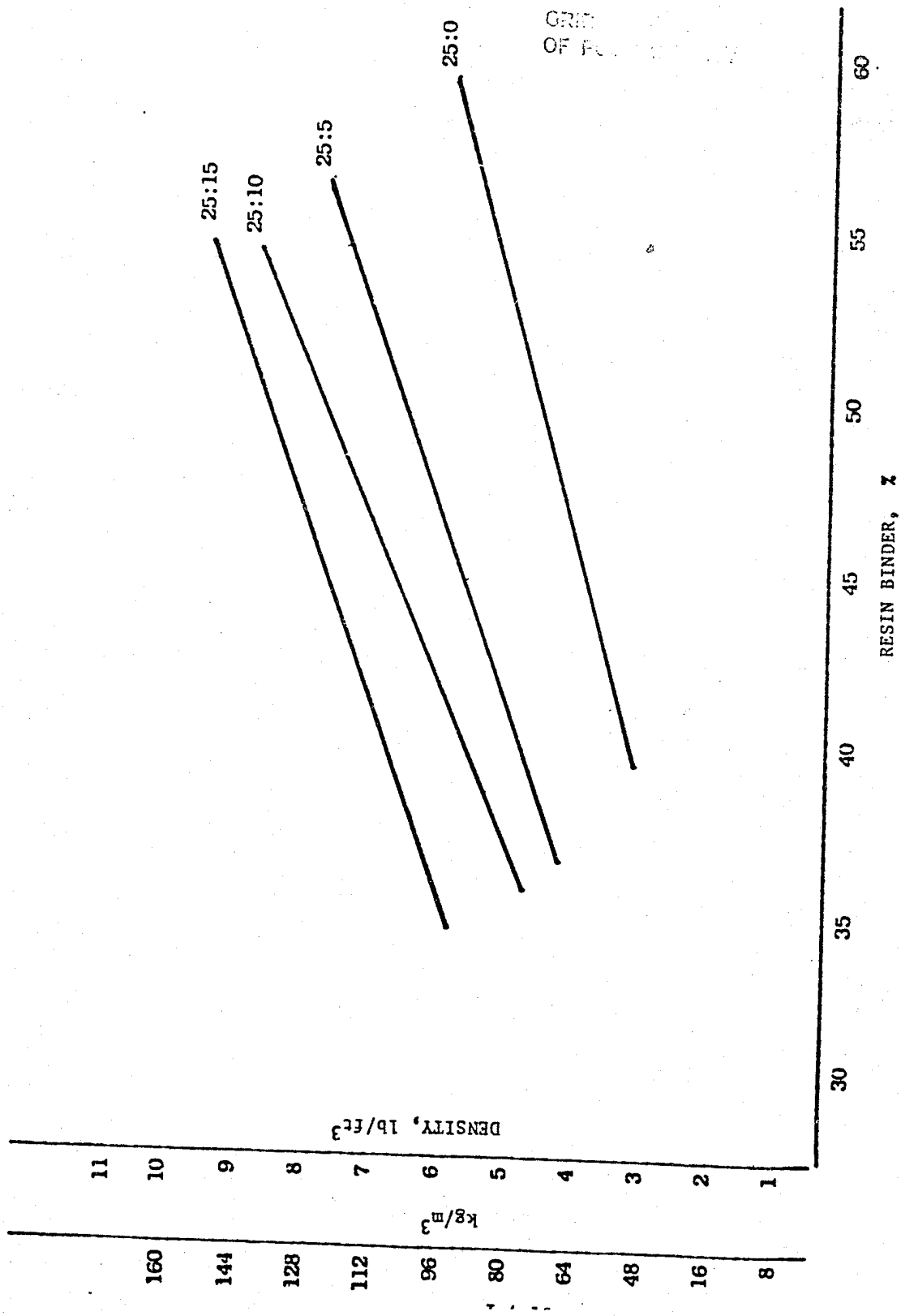


Figure 4-6 Effect of resin loading on density of closed-cell foams produced at ratios of macroballoons to glass microballoons of 25:0, 25:5, 25:10, and 25:15, respectively.

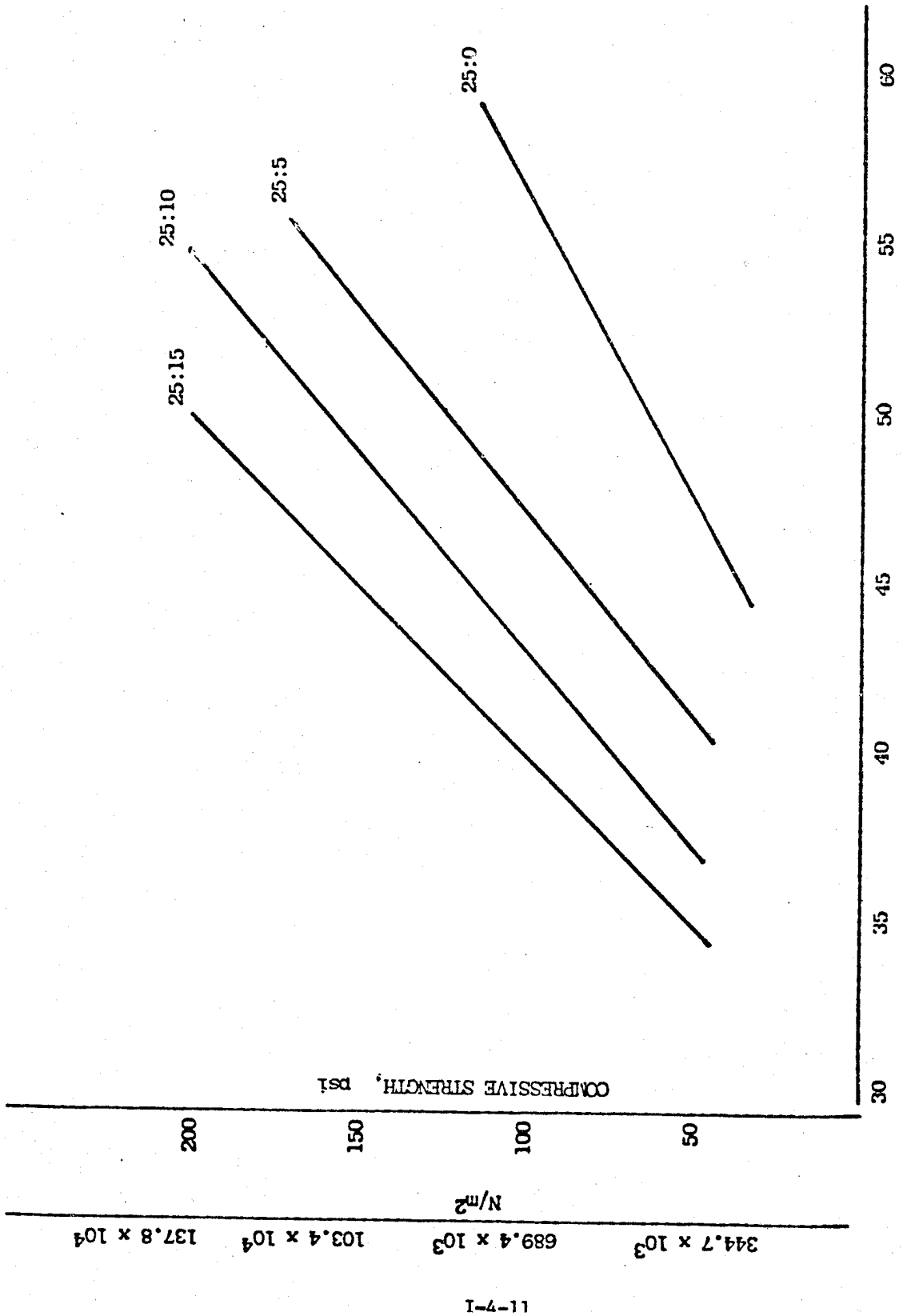


Figure 4-7 Effect of resin loading on compressive strength of closed-cell foams produced at ratios of macroballoons to glass microballoons of 25:0, 25:5, 25:10, 25:15, respectively.

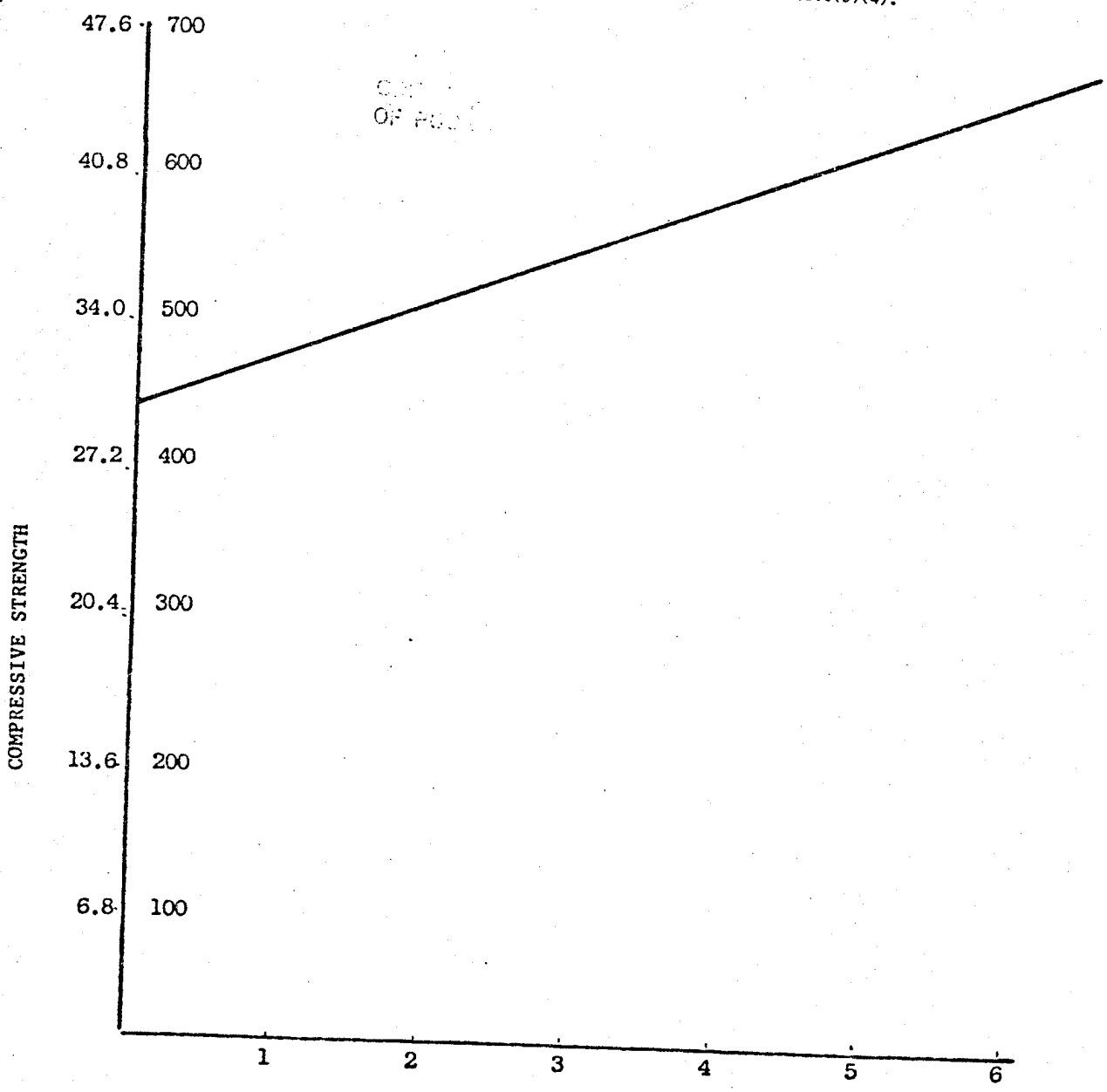


Figure 4-8

Effect of glass fibers on the compressive strength properties of honeycomb.

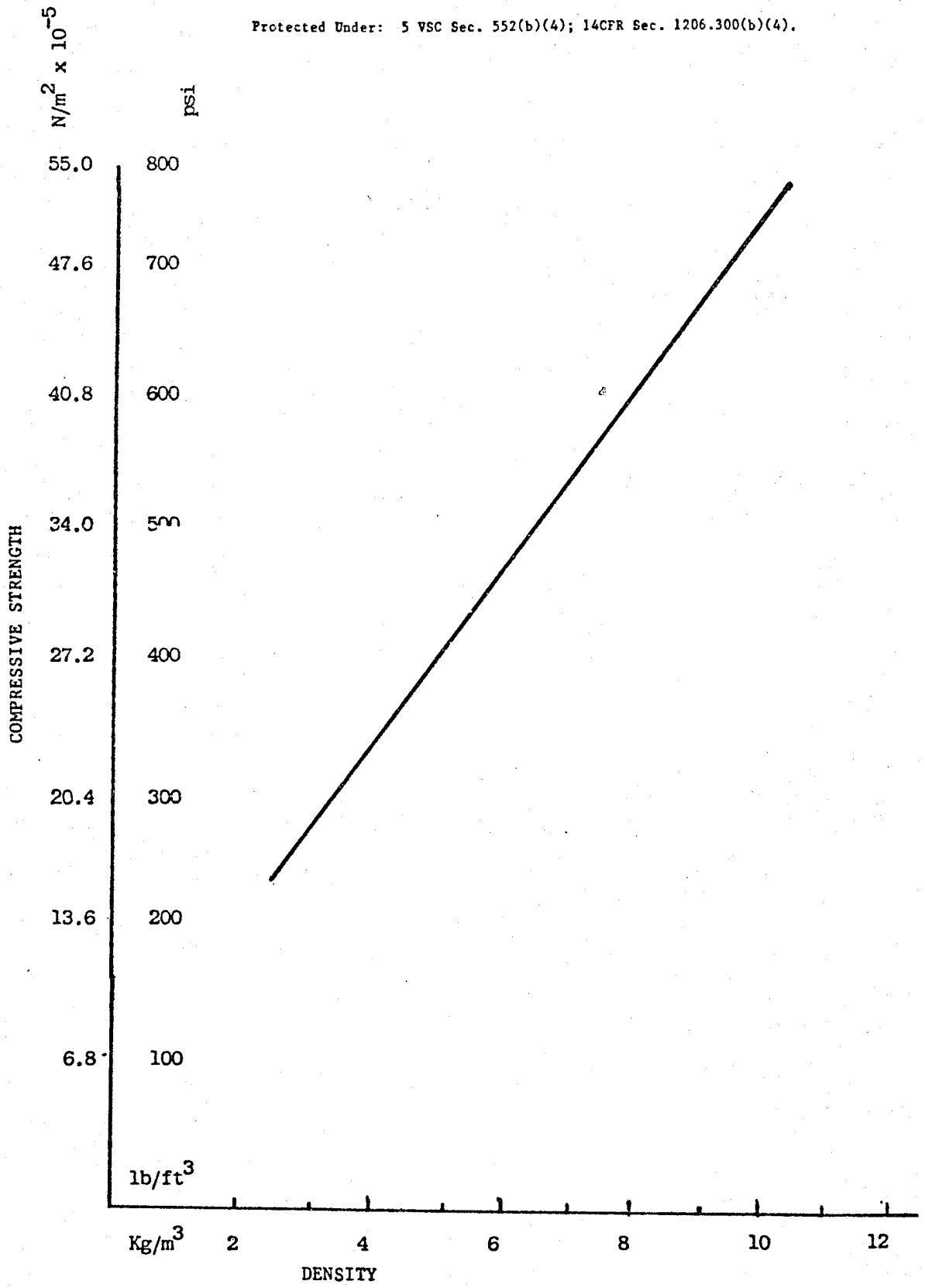


Figure 4-9

Reinforced Honeycomb, Density-Compressive Strength Relationship.

RESULTS FROM WEBER AIRCRAFT

Table I

	Sample #1	Sample #2
Shear Strength N/m^2 (psi)	3.4×10^5 (49)	3.9×10^5 (56)
Compressive Strength N/m^2 (psi)	6.6×10^5 (96)	7.5×10^5 (109)
Density kg/m^3 (lb/ft^3)	72 (4.5)	84 (5.3)

The final candidate selected for cargo bay floor core applications was produced by reinforcing a low density Nomex honeycomb with polyimide resin.

The galley and cargo bay floor core materials produced in the preceding section were then evaluated for all physical, mechanical and thermal properties. The resulting data was used to generate a final specification for each of the syntactic foams. The final specifications are reported in Table II for galley core material and in Table III for cargo bay floor core material.

GALLEY CORE MATERIAL

Table II

PROPERTY	TEST METHOD	UNITS	SPECIFICATION
Weight	D-1564	kg/m^3 lb/ft^3	88.0 5.5 max
Compressive Strength	MIL-STD-401	N/m^2 psi	6.2×10^5 90 min
Shear Strength	C-273	N/m^2 psi	3.4×10^5 50 min
Oxygen Index	D-2863	% oxygen to sustain combustion	42 min
Smoke Density	E-662	D_S	30 max
Thermal Degradation		Weight Loss at 400°F 204°C	2% max

CARGO BAY CORE MATERIAL

Table III

PROPERTY	TEST METHOD	UNITS	SPECIFICATION
Density	D-1564	k/m^3 lb/ft^3	96 6 max
Compressive Strength		N/m^2 psi	4.1×10^6 600 min
Oxygen Index	D-2863	% oxygen to sustain combustion	45 min
Smoke Density	E-662	D_S	20 max
Thermal Degradation		Weight Loss at 400°F 204°C	2% max

4.6 Task VI - Sample Preparation

A total of six panels, having dimensions of approximately 61 x 91 cm (2 x 3 ft), were produced for each of the selected galley and cargo bay material specifications and delivered to NASA LB Johnson Space Center.

The material met the requirements established in the preceding section.

PART II

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Part II

1

PROGRAM SCOPE AND OBJECTIVES

PART II

This program was undertaken to select foam compositions and to investigate an apparatus to produce polyimide foams using foam-in-place techniques.

A major objective of this program was the fabrication of prototype samples and sufficient quantities of the composition or compositions for evaluation by NASA-LB Johnson Space Center, Houston, Texas.

The program consists of five tasks which define the general objectives of the work plan. The work plan constituting the various tasks of the program and their milestones is shown in Figure 1.

Task I covers the design and fabrication of a spray gun apparatus for use in spraying a semi-fluid mixture of polyimide composition. This process produces a syntactic foam which is curable at room temperature or by application of heat.

Task II and III cover the modification of the apparatus described in Task I or the design and fabrication of a heated spray gun apparatus to spray polyimide resins in order to achieve foaming and curing in a single step process.

The products derived from the study of these different systems are evaluated and the final candidate selected in Task IV for submittal to NASA-LB Johnson Space Center as proposed in Task V.

A final task dealing with Reporting and Coordination covers the effort necessary to report the program status and includes a mid-term and one final presentation to acquaint NASA-LB Johnson Space Center technical personnel with the progress of the program.

The overall technical content of the program covers a period of twelve (12) months starting with July 15, 1983.

PROGRAM SCHEDULE

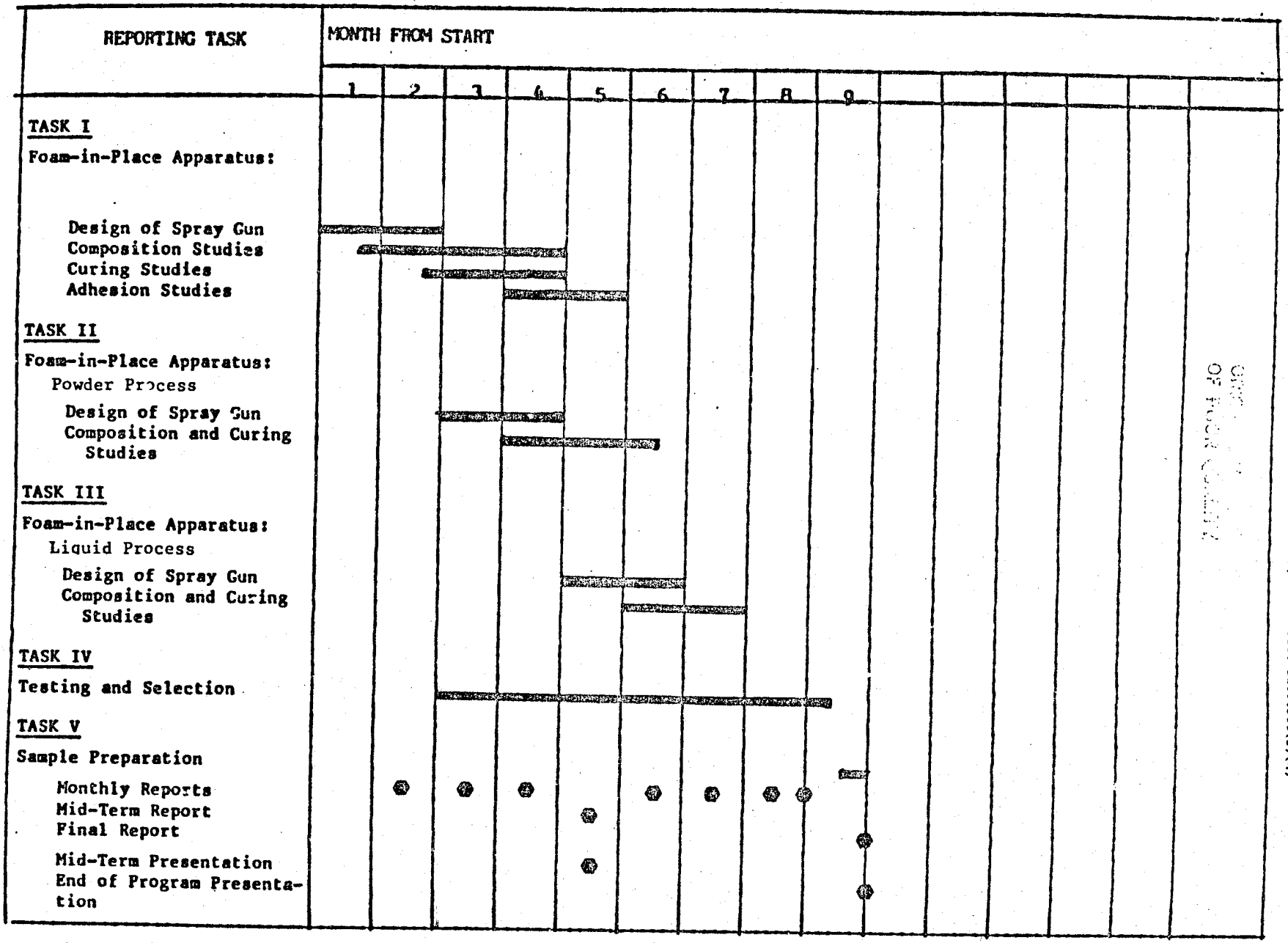

Figure 1 MILESTONE CHART

2

PROGRAM APPROACH

The investigations leading to the selection of methods to produce foam-in-place polyimide foams follow the approaches described in the previous sections and utilize polyimide resin systems previously developed.

The approach to the development of foam-in-place polyimide foam was planned to proceed with the design and development of a spray apparatus for spraying a mixture of resin which upon curing produced a foam.

Attempts have also been made to modify the same apparatus by using a heat source for spraying, foaming and curing the resins on metallic substrates.

3

EXPERIMENTAL PROCEDURES

This section outlines the experimental procedures used to produce the final foams.

3.1 Foam-In-Place Procedures

The first foam-in-place method evaluated in this program involved the use of the microballoons. In this process the microballoons were first mixed with a binder and the mixture sprayed using the spraying apparatus shown in Figure 2. The composition was sufficiently tacky to bond to all the metallic substrates proposed.

The foam obtained using this process possessed a density of 64-80 kg/m^3 (4-5 lb/ft^3).

Foam-in-place experiments using other polyimide resins were also undertaken. The apparatus used consisted of a hot air gun operating at an air pressure of approximately 10.3×10^4 N/m^2 (15 psi) and an air temperature of approximately 537.7°C (1000°F) at 10 cm (4 inches) from the gun.

The resin was fed slowly into the hot air stream. The resin foamed immediately and the hot foam was propelled onto the metal surface of the substrate. Adhesion to the substrate was good.

This type of foam possessed a density of 8-16 kg/m^3 (0.5-1.0 lb/ft^3) and a non-homogeneous cellular structure.

The hot air gun is shown in Figure 3. The unit is a Sylvania hot air heater which can produce air temperatures up to 593°C (1100°F). A Wilkerson air pressure regulator is used to control the air pressure leading into the Sylvania heater.

Foam-in-place experiments with the liquid resins were not easily carried out due to the presence of the solvent that interfered with the foaming process.

FOAM-IN-PLACE APPARATUS

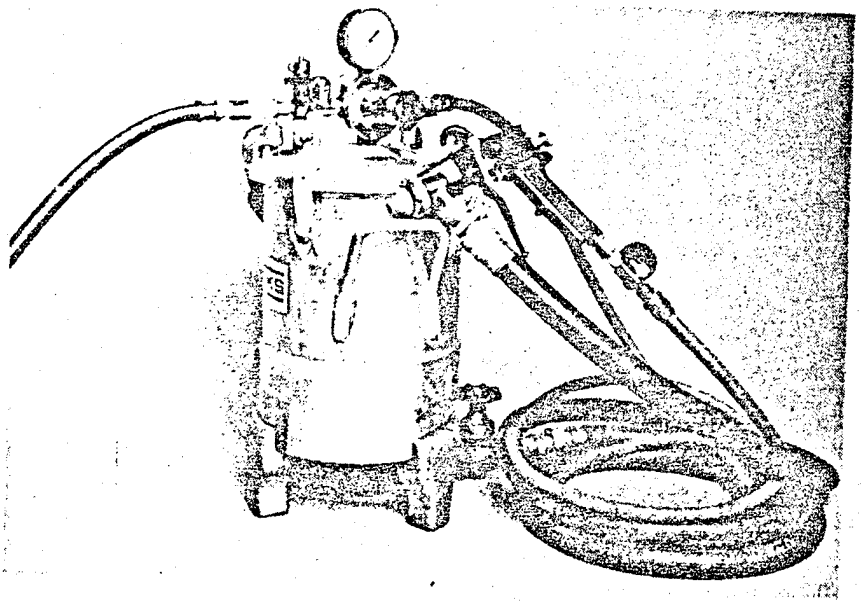

Figure 2
The Binks two gallon pot and spray nozzle

FOAM-IN-PLACE APPARATUS DEVELOPMENT

PRELIMINARY TESTING WITH SYLVANIA FORCED AIR HEATER

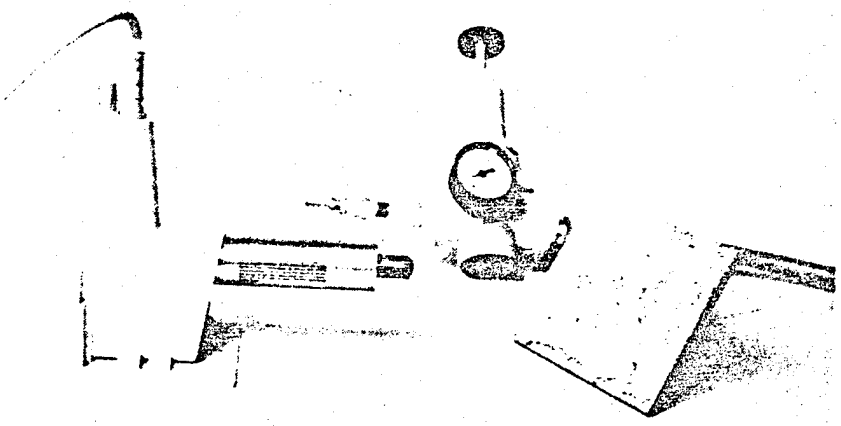

Figure 3

The Sylvania forced air heater and air pressure regulator used to foam-in-place the foam shown on stainless steel.

4

EXPERIMENTAL RESULTS

This section covers the work carried out to investigate and select processes to foam-in-place polyimide resins. The section starts with the development of a foam-in-place apparatus to produce closed-cell foam by spraying a syntactic composition (4.1) followed by the development of a heated spray gun apparatus to foam-in-place polyimide resins (4.2 and 4.3).

4.1 Task I - Foam-In-Place Apparatus Development

In the present program selected microballoons were blended with a liquid resin binder and the mixture was then sprayed onto the surface of a metallic substrate.

The process of producing foamed-in-place syntactic foams involved evaluation of:

Spray techniques
Viscosity of the syntactic composition
Adhesion to metallic substrate

4.1.1 Task I - Design of Spray Gun

Preliminary studies carried out with a conventional paint spray gun suggested that spraying the syntactic composition could be accomplished with a single component spraying system specifically designed to deliver high viscosity, materials.

A Binks spray gun system was selected for this application and was shown in Figure 2. The Binks spray gun system consists of a 2 gallon pressure pot with a pressure regulator and a wide mouth nozzle spray gun which is also equipped with a pressure regulator. The mixture of microballoons and resin was charged into the pressure pot and delivered at high pressure through the spray gun for deposition on selected substrates.

4.1.2 Task I - Composition Studies

The compositions used to produce the foams were prepared by blending various components specifically designed to add flame resistance and low smoke to the final product with a fluorocarbon binder.

The first determination involved the effect of viscosity on spraying. The high rate of drying caused by the air atomization produced starved foams, therefore, composition was modified by increasing the solvent content. A high solvent content was found to be necessary to accomplish lay-up of the syntactic composition on the substrates. The optimum pot pressure was found to be between 172.3×10^3 N/m^2 (25-30 psi) and the optimum nozzle pressure about 124×10^3 N/m^2 (18 psi). The total time required to deliver two gallons of mixture continuously was two minutes.

4.1.3 Task I - Curing Studies

In the execution of the present task room temperature curing was accomplished in all cases after the composition was sprayed on the metallic substrates which were held in vertical position. No running of the resin was noticed and the mixture cured to a hard foam. The foam produced by this process possessed density characteristics in the range of 112-128 kg/m^3 (7-8 lb/ft^3) a compressive strength of $103 \times 10^3 - 172 \times 10^3$ N/m^2 (15-25 psi) and a tensile strength of $55 \times 10^3 - 68 \times 10^3$ (8-10 psi).

The foam did not burn and possessed a limiting oxygen index in the range of 45-55. The evolution of smoke was determined using the NBS smoke chamber and was found to be 25. The thermo-chemical properties of the foam are defined by the TGA thermagravimetric analysis curve.

The foam composition did not cause pitting of aluminum, steel or titanium.

4.1.4 Task I - Adhesion Studies

The adhesion characteristics of the foam composition on carbon steel, aluminum and titanium alloys were found to be strong and the foam was not easily damaged.

The metal substrates were prepared as follows: a) wiping with acetone and allowing to dry, b) by first coating the substrates and c) by cleaning the surface with a paper towel to remove any loose particles. The foam composition was applied with the Binks spray-up equipment and the resulting foam was tested for cure and adhesion.

No difference in the adhesion properties of the foam material was found to exist between the three metal substrates or the method of substrate preparation. The foam was structurally strong and not easily damaged.

Figure 4 shows three head-on views of polyimide foam mixtures foamed-in-place with the Binks' system on steel, titanium and aluminum.

4.2 Task II - Foam-In-Place Apparatus Development Powder Process

It was found that this process requires the use of a heat source to start the foaming process. Once foaming is initiated sufficient thermal energy is available for the subsequent curing step.

The key design parameters determined from the preliminary experiments were: the hot air temperature and velocity, the rate of powder delivery, and the distance between the hot air nozzle and the metallic substrate.

The optimum hot air temperature was found to be between 371-482°C (700-900°F) at a velocity between 90-150 m/minute (300-500 ft/m). The maximum rate of powder resin delivery into the hot air streams investigated was approximately 20 g/minute (.04 lb/m). A higher rate of powder delivery may be possible with

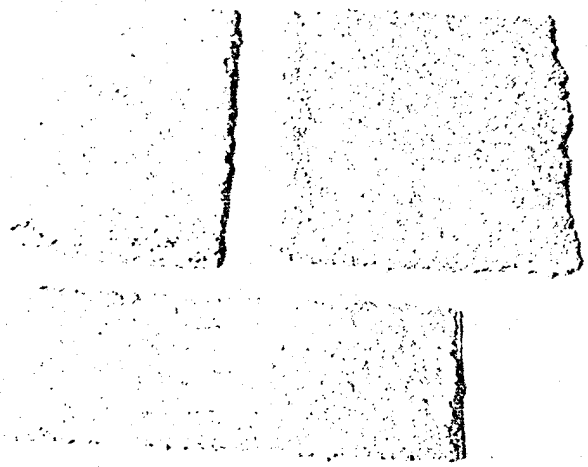

Figure 4

Head-on view of syntactic foam mixture foamed-in-place with the Binks system on carbon steel, titanium and aluminum.

a spray gun system similar to the Binks system described above. The optimum distance between the hot air nozzle and the substrate is approximately 10-15 cm (4-6 in) for the system shown in Figure 3 and 30 cm (12 in) for the system shown in Figures 5 and 6.

These preliminary experiments were carried out using the heat gun, as described below.

4.2.1 Task II - Design of Spray Gun

The heat gun-air pressure apparatus was shown previously in Figure 3.

The gun is an infrared heater, 30.5 cm (12 inch) long which heats air to 593.3°C (1100°F). The heating rate is controllable over the operating range by varying the air flow through the heater. The air flow provides the medium for transfer of the powder or the foamed material to the substrate.

This highly efficient, although small flameless heater was used for the preliminary experiments to prove the concept of foaming.

The experiments were carried out at the lowest permissible air pressure (the gun will not operate below 68×10^3 N/m^2 (10 psi)) of 82.7×10^3 - 103.4×10^3 N/m^2 (12-15 psi) by feeding the powder in the hot air stream at a rate of approximately 20 g per minute. The powder melted, expanded into a foam and deposited on a vertically mounted aluminum plate. The resulting foam possessed an irregular cellular structure

Figure 7 shows a typical foam-in-place sample produced from the Sylvania system.

Based on these preliminary experiments a heater spray gun apparatus with high thermal energy was considered to be the best choice for this application. To further prove this concept a series of experiments were carried out with a commercial space heater. The commercial space heater used shown in Figure 5 is a 150,000 Btu/hr forced air propane gas heater. This unit delivers 0.2 m^3/s (450 cfm) of hot air with an exit temperature of 1018°C (1864°F).

To lower the heat flux produced by this unit we attached a conical section of 1/16 inch carbon steel with a maximum diameter of 43 cm (17 in). The modified space heater is shown in Figure 6. This modification lowered the hot air velocity from 305 m/min (1019 ft/m) to 85 m/min (285 ft/m) and the exit temperature to 315°C (600°F).

Even at these conditions the powder resin did not produce a self adhering, strong foam suitable for insulation of systems.

4.3 Task III - Foam-in-Place Apparatus - Liquid Resin Process

Because of the flammability of the solvent preliminary experiments were carried out with the Sylvania heating system.

Universal Model 150 FA Propane Heater

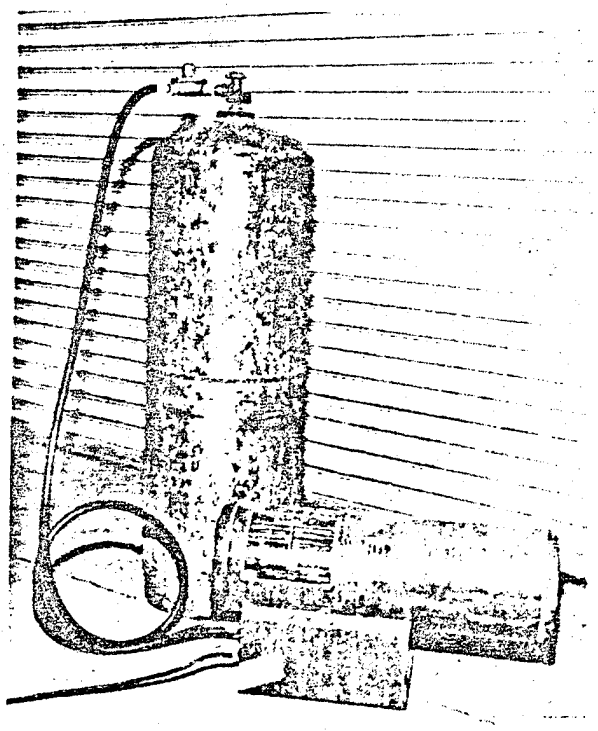

Figure 5

The Universal forced air propane gas heater.

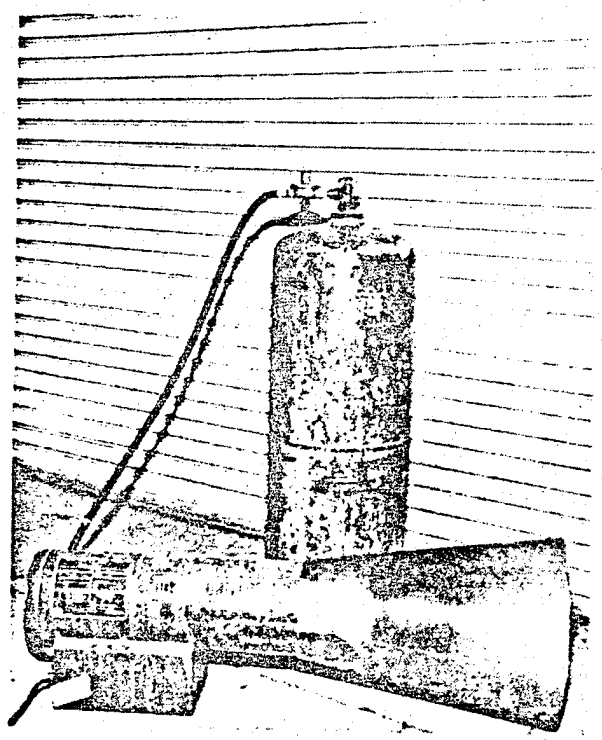

Figure 6

The Universal forced air heater with conical section used to reduce the heat flux.

Figure 7

Head-on view of powder resin foamed-in-place with the Sylvania system.

Flashing of the solvent was experienced which made this process difficult to carry out even on a small scale. Due to these difficulties the liquid resin process was rejected.

4.4 Task IV - Testing and Selection

The most important objective of this task was to select an apparatus to foam-in-place a polyimide resin meeting the requirements of the program. The process selected was that based on microballoon-resin system using a spray gun to deposit the syntactic composition. The end product of this process has the properties reported in the following table together with the goals of the program.

Table I

Material Property Specifications for the Foam-in-Place Composition selected vs. the Program Goals.

PROPERTIES	PROGRAM GOAL	RESULTS
Density	80 kg/m^3 (5 lb/ft^3)	136 kg/m^3 (8.5 lb/ft^3)
Tensile Strength	34 x 10^3-654 x 10^3 N/m^3 (5-95 psi)	62 x 10^3 N/m^2 (9 psi)
Compressive Strength	110 x 10^3 - 413 x 10^3 N/m^2 (16-60 psi)	144 x 10^3 N/m^2 (21 psi)
Service Temperature	-78°C to +274°C (-110°F to + 525°F)	-78°C to + 232°C (-110°F to +450°F)
Corrosion	No Pitting	No Pitting on Aluminum or metal
Limiting Oxygen Index	40	50
Smoke Density	30 - 50	25
Thermo-Chemical Properties	No loss below 204°C (400°F)	less than 1% (water)

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Ref. 2, Gagliani, J., Sorathia, V.A.K. and Wilcoxson, A.L., "Development of Fire-Resistant, Low Smoke Generating, Thermally Stable End Items for Aircraft and Spacecraft", Contract NAS 9-15050, National Aeronautics and Space Administration, Houston, Texas, Final Report 1977.

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